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River dynamics and nanopaticles formation: A comprehensive study on the nanoparticle geochemistry of suspended sediments in the Magdalena River, Caribbean Industrial Area

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ABSTRACT

The coastal zones on continental shelves are the main channels for the distribution of fluvial-sourced suspended sediments (SSs). In the current research, the monthly average amount of SS draining into the Caribbean Sea from the Magdalena River in northern Colombia was analyzed to detect nanoparticle (NPs) containing potential hazardous elements (PHEs). The ecological authorities of Colombia claimed that the climate change is the key reason behind land erosion and floods occurred in the last years; therefore, an elaborate understanding of NP dynamics between the Magdalena River body and streambed is an essential issue in SS research. In this work, the NP geochemistry of SS in the Magdalena River estuary was studied from the perspective of water quality controls on SS sorting. The morphologies and the structures of NPs (<100 nm) were examined by field emission scanning electron microscopy (FE-SEM), high-resolution transmission electron microscopy (HR-TEM), and selected area electron diffraction (SAED)/micro-beam diffraction (MBD)/energy dispersive X-ray spectroscopy (EDS) techniques. The average size of NPs was found to be greater than 2 nm and Al, Ti, Fe oxides, and other hazardous elements were also detected in the SS. The obtained data confirmed that these typical categories of NPs caused the occurrence-dependent intensification of a conjugative transmission rate associated with the regulators. The advanced electron beam technique provided a clear insight into SS transportation; therefore, it could be used as an essential instrument for river supervision/dynamics.

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1. Introduction

Suspended sediment (SS) passage plays an important role in global geochemical cycles of the natural environment (Respetro et al., 2018; Tejeda-Benitez et al., 2016; Nordin et al., 2018). Recent experimental studies reveal that SS loads of many rivers throughout the world have been significantly disturbed due to climate change, land use change, tillage, mining, soil and water preservation, and other several human activities (Tang et al., 2018; Tuset et al., 2016; Wall et al., 2015).

Most of the earlier studies mainly concentrated on investigating the effect of nanoparticles (NPs) on potential hazardous elements (PHEs) leaching to the soil and atmosphere (Nordin et al., 2018; Civeira et al., 2016); however, few of them reported that SS contains more NPs. Anthropogenic activities play a conspicuous role in mobilizing SS in the Madgalena River (Respetro et al., 2018). The presence of excessive ultrafine NPs in the source river increase the cost of drinking water management (USEPA, 2000). The accumulation of ultrafine NPs in water (referred to as siltation) profoundly contaminates the environment (Park and Hunt, 2018). Particle size, which is a traditional feature, is constant in association with SS translocation; therefore, it can be used to infer the dynamic nature of SS assembly as well as to comprehend the sorting mechanism related to SS translocation (Tang et al., 2018).

Nanoparticles transmit substantial quantities of toxins,

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comprising the major portion of hazardous compounds by weight (Li et al., 2003). Industrial activities generate nano-sized particles as accidental waste products, which eventually capture other harmful elements in nature (Ribeiro et al., 2010; Ramos et al., 2017). Despite the health, safety, and environmental risks posed by the release of nanoparticles from river sediments, there are limited studies in the literature on the subject.

Wu et al. investigated the effects of fluvial processes on tropical estuaries. The change in the Magdalena River discharge system has significantly affected the salinity dissemination and the SS deposition cycle. Suspended sediments are created by the multifaceted relation among fluvial sources and geophysical progressions at different time and space scales (Civeira et al., 2016; Restrepo and Escobar, 2018); therefore, they can be classified as river-dominated SS dispersal basins (Hanebuth et al., 2015; Walsh and Nittrouer, 2009). In this study, the temporal trends of Magdalena SS load were analyzed on a basin scale and the effects of NPs and human activities on estuarine movement and SS translocation was studied in detail.

The main aims of this study were to (1) discover the relationship between NPs and PHEs in SS and (2) determine the impacts of NPs on PHE proportion in the Magdalena River water phase and speciation circulation in SS. This is a pioneer work reporting the effects of NPs on the mobility of PHEs in SS. The obtained results provided a foundation for revealing the ecological risks caused by NPs in SS; therefore, our experiment could be a good reference for water conservation in the estuarine system.

2. Materials and methods

2.1. Study area

Fluvial SS is an important constituent of any river and is also important to nutrient transference and permanence of river bodies and living microorganisms (Restrepo and Escobar, 2018). The change in SS due to climate change or human activities has significant influence on fluvial areas, as well as on deltaic and estuarine zones, thus resulting in variation in littoral environment and diminution in fish abundance (Gao et al., 2011). Therefore, maintaining regular information on SS loads in coastal areas is indispensable in order to control the harmful effects produced by climate change and human activities (Syvitski and Kettner, 2011). Moreover, the rapid destruction of coral banks and oceanic grass in the Caribbean environment due to the presence of SS contaminants in the Magdalena River has become an alarming concern (Moreno et al., 2015).

The Colombian Caribbean plain is located in the northernmost area of South America (Fig. 1), and the Magdalena River (with an extension of 1540 km and drainage of $257.438 \times 10^3\ km^2$) is the main river in this region. The Magdalena River covers 24% of Colombia (Restrepo et al., 2018), hence, it is the main contributing source of SS to the Caribbean Ocean (Higgins et al., 2015). The rate of deforestation has been significantly reduced over the last years (Etter et al., 2006), and its impact on SS transference rate is evident (López et al., 2017). The SS load in the Magdalena River between 2000 and 2010 was assessed as $16\ Mt\ yr^{-1}$ (Restrepo et al., 2015).

2.2. Sampling strategy

In Colombia, the analysis of SS loads and their underlying developments, such as SS yield, SS delivery ratio, is still in an initial phase (López et al., 2017). The present sampling and analysis work was started to collect information on NP geochemistry of SS in the Magdalena River by considering high fluvial liberation, and unusual seasonal circumstances. Our work was inspired by previous research that reported the relationship between NP loading rate and movement rate in gravel SS beds (Nordin et al., 2018; Civeira et al., 2016).

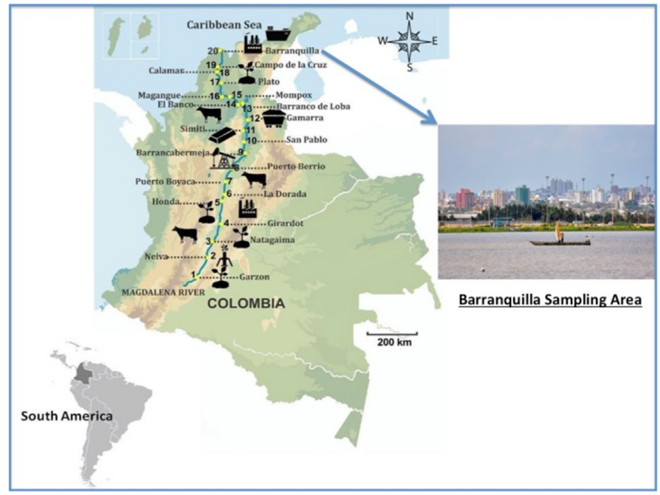

Fig. 1. The area of Magdalena River basin.

The SS samples were collected monthly for an entire hydrological year spanning from April 2017 to March 2018 at Magdalena (in the Barranquilla City) in the central reach by a time-integrating SS trap (Phillips et al., 2000). A total of 36 (3 per month) samples were collected at the gauging positions closest to the Magdalena River mouth. A crucial benefit of these SS traps as compared to other sample collection techniques, such as work-exhaustive technicians (Wren et al., 2000), was the ease of compilation of time-combined SS per unit time, hence, the logistical difficulties of retrieving numerous SS snapshots from a given position to collect real data on SS geocharacteristics was easily avoided (Massoudieh et al., 2012). In order to obtain correct results from these time-integrating testers, the nonexistence of airlocks during enclosure into the SS as well as debris in the inlet tube was ensured. The 4-h integrated SS samples were collected using a transportable centrifugal sampler (Cao and He, 2013) at the outlet of the Magdalena River, and the samples were applied to depict spatial designs of particle magnitude in SS. Transportable centrifuges offered a consistent means of collecting symbolic time-integrated SS at areas where the disposition of Phillips traps was more challenging, for example, where the depths of sediments were inaccessible. The SS samples were studied to describe the nanomineralogy of SS particles by assuming that all sediments were created from a single flood incident. A similar sampling procedure was previously reported by Schubert et al. (2012) and Masson et al. (2018).

2.3. Analytical methods

The geochemical configuration of SS samples were examined by X-ray powder diffraction using a Phillips PW1830 diffractometer with Cu Kα radiation and a scan range from 2 to 60° 2 h, Raman spectroscopy using a Renishaw-model Invia Reflex Raman system, operated in the confocal mode (Ribeiro et al., 2010). Most of the Raman analyses were performed on polished epoxy-bound pellets used for petrographic analysis. Spectra obtained from representative SS particles were deconvoluted in order to calculate the Raman parameters and determine the precise frequencies, bandwidths, and the relative intensities of the bands of the carbon materials. Field emission scanning electron microscopy (FE-SEM) fitted with a turbo pumped chamber, a motorized stage, an Oxford energy-

dispersive X-ray (EDS) spectrometer having resolution N133eV, and a four-quadrant back-scatter detector, was used to identify the minerals by conventional SEM techniques, based on observation of whole-specimens using natural and/or polished surfaces, high-resolution transmission electron microscope (HR-TEM, 200 kV) equipped with an efficient FE cathode and an energy omega-filter selected area electron diffraction (SAED) and Fast Fourier transform (FFT), scanning transmission electron microscopy (STEM) micro-beam diffraction (MBD) and energy dispersive X-ray spectroscopy (EDS) techniques (Ramos et al., 2017; Quispe et al., 2012). The elements with high atomic number and low atomic number were found in the brightest zones (Silva et al., 2011a,b,c) and the dark-field zones of the TEM image, respectively (Silva et al., 2011d,e); therefore, it confirmed the presence of PHEs and NPs in SS samples. A poorly crystalline silica phase (resembling but not equivalent to tridymite) was used as part of the Siroquant analysis procedure for the ash samples to estimate the percentage of non-crystalline (amorphous) material present.

3. Results and discussion

3.1. River dynamics and nanopaticles formation

The Magdalena River movement of separate tidal cycles was mostly in the on-offshore course. The tidal effect is also reproduced in the temporal variations in the SS outline in the inferior part of the Madgalena River water column. High standards are linked to the time of spring tide both proximate to the ocean bed and above the ocean bed. Temporal alterations in the increasing SS transportation in the inferior Magdalena River water column had designs comparable to those of the river movement. The spatial and the temporal tendencies in total particle size composition of Magdalena SS is important to understand SS mobilization and allocation within blocked reaches and SS sorting in the water-level fluctuation area. The SS assembles in the water column and the occurrence of the constant layer at the mid-depth a profound influence on the SS dynamics of the Magdalena River transportation structure.

In this study, the spatial and the temporal patterns of total particle size composition of Magdalena River SS are studied using the descriptive time-integrated method, and the SS sampleds, were collected at upstream watercourse outlets as well as along the reservoir during the rainy season. SS confirmed a perfect longitudinal fining tendency, and the inflow of SS from upstream watercourses evinced that SS contribution in the rainy period was greater as compared to the dry period (Tejeda-Benitez et al., 2016). The SS samples mainly originate from upstream main watercourses during the wet period, and thus SS loads were released from SS assemblies during major rainstorm (Tang et al., 2018).

The natural issues and the anthropogenic influence both can disturb the SS dynamics of a river (Tuset et al., 2016). The study of NP deposition and invection from canal beds is important for ecological modelling. The uncontrollable deforestation, the rapid industrial development, and the illegal increase in agronomy, pasture, and mining industries have destroyed the normal hydrographs of the Magdalena River (Tejeda-Benitez et al., 2016). A high percentage of habitats of Colombia (approximately 38 million inhabitants) including Bogota, Medellin, Cali, and Barranquilla stay in the Magdalena River division, thus they majorly depend on this river. Most of the municipalities in Colombia have no proper wastewater management plant (Baron et al., 2013), thus the direct ejection of industrial sewage and polluted trashes from coal easily contaminate the Magdalena basin.

The crystalline compounds identified by the various techniques in the SS include carbonates, hydroxides, oxides, phosphates, silicates, sulphates and sulphides. The phase transition reactions associated with formation of these materials in the burning waste piles represent a highly dynamic process. Quartz and silicates can be incorporated to the sediments by erosion of meta-morphic and sedimentary rocks. The microscopic analysis was adopted to reveal the clay composition of the Magdalena River, and by EDS technique, illite, smectite, illite/smectite, kaolinite, and quartz were identified as major minerals of this river. The morphologies of these minerals were examined by SAED/FFT and HR-TEM methods. Moreover detrital quartz, Fe-oxides, oxy-hydroxides, and diatom skeletons were also noticed in the Magdalena clay. Diatom frustules were comprised of amorphous Si-compounds, thus they were more reactive than silicates and quartz. In order to understand the weathering process of industrial sludges as well as to interpret the influences of the environment and Fe minerals on SS loads of the Magdalena River, the atomic structures of aluminum, iron, and silicon were examined by FE-SEM and HR-TEM.

The detection of natural and anthropogenic organic matters (OMs) in SS can improve the geostability and the bioavailability of NPs (Nordin et al., 2018; Silva et al., 2011a). The higher NP accumulation rate in OMs can result in high adsorption capability of extracellular materials in SS.

In the present study, the development of iron-aluminosilicates is clearly recognized; hence, a detailed investigation needs to be carried out in future studies to understand the effects of such iron-aluminosilicates on river erosion.

The intensities of NP and ultrafine particles (Figs. 2–7) in the SS bed of the Magdalena were comparable. The detected ultrafine particles and NPs entered the pores of the SS bed by percolation and this phenomenon was detected by a specific filtration factor, α. In addition, as NPs started to accumulate in the SS bed, the size of pore area is constantly reduced simultaneously, thus decreasing the filtration rate of NPs in the SS bed.

Furthermore, illite with halite (Fig. 2A), quartz (Fig. 2B), halite with amorphous Al–K–Fe–Si-particles (Fig. 2C), barite (Fig. 2D), OM containing amorphous Al–Si–K particles and Ti dioxide (Fig. 3A), zircon (Fig. 3B), kaolinite, anatase, rutile, chlorite, k-feldspar, plagioclase, calcite, and Ca–Al–Fe-sulfates were also found in SS. In general, NPs enriched with quartz had fewer amount of PHEs as compared to clays. The investigated NPs in SS manifested diverse forms of colloids. Hence their large superficial area per unit mass turned them into superb binding minerals for other organic/inorganic pollutants.

The obtained date presented the greatest significant mode of nanomineralogy co-variability (40.4%) among SS properties was the

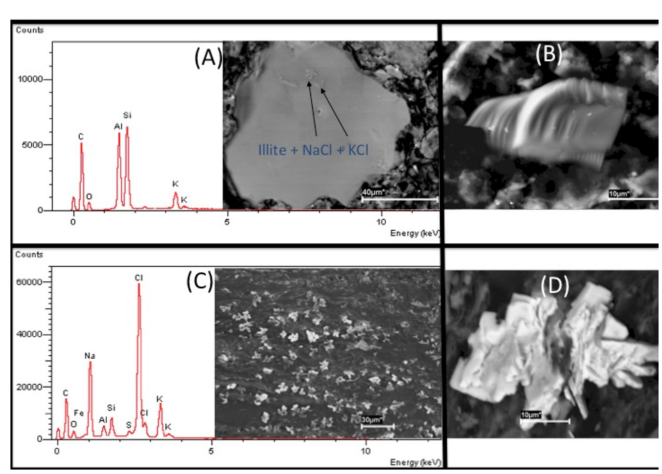

Fig. 2. Selected figures of the illite with halite (2A); Quartz (2B); Halite with amorphous Al–K–Fe–Si-particles (2C); Barite (2D).

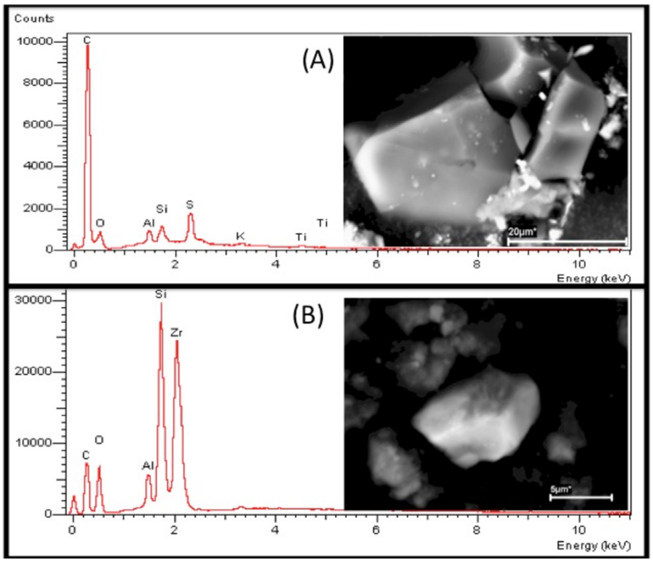

Fig. 3. Organic mater containing amorphous Al–Si–K particles and Ti dioxide (3A); zircon (3B).

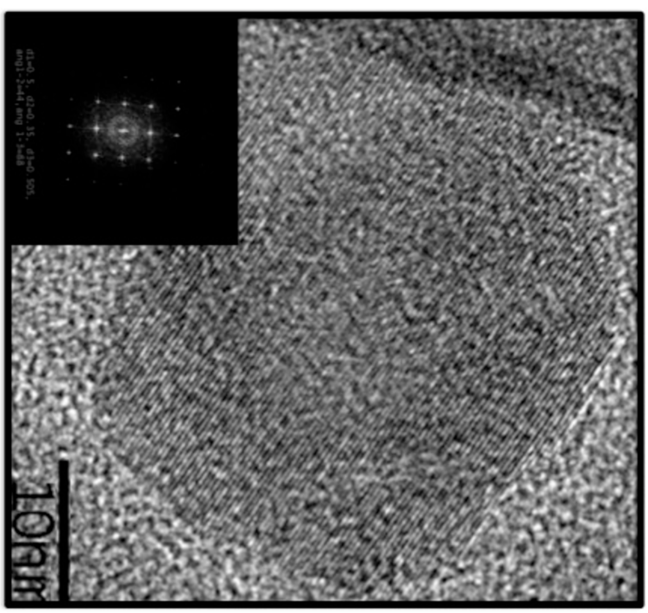

Fig. 4. Illustration of nanokaolinite.

separation of nanoclay-sized particles (e.g. Fig. 4) form grainier NPs due to the hydraulic organization in the SS transportation course. It was observed that the natural NPs were composed of carbonaceous, inorganic, or microbial compounds. Due to their large specific surface area, NPs were efficient sorbents of PHEs.

3.2. Potential hazardous elements (PHEs) and association with NPs

Trace elements such as Al, Cr, Cu, K, Mg, Mo, U, V, Zn, and Sn were relatively enriched in the studied microparticles. In contrast, As, Ni, W, Pb, Ba, and others hazardous compounds are significantly enriched in the nanoparticles. The rare earth element (REE) constituents such as La, Ce, Pr, Nd, Sm, Eu, and Gd are slightly enriched in several detected nano-clays. The main mode of occurrence of these elements were as phosphates (e.g. monazite). The

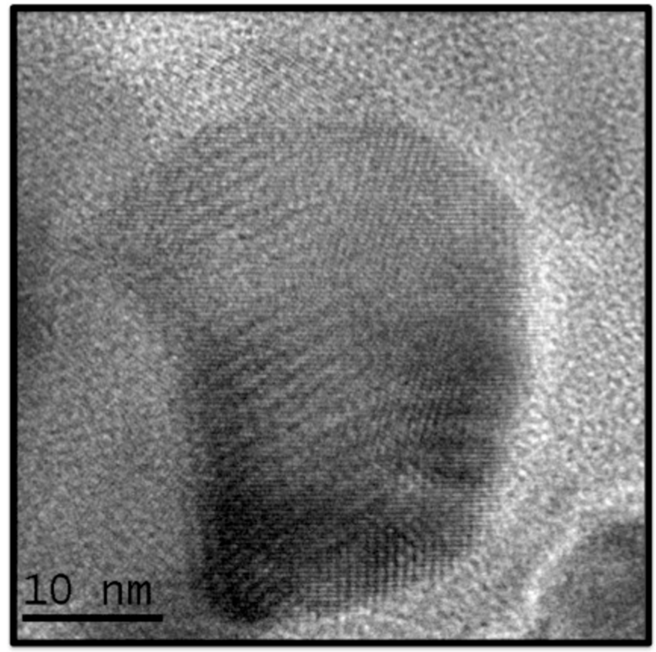

Fig. 5. Fe-nanohematite containing Cd, Co, Cr, Cu, Hg, and Pb.

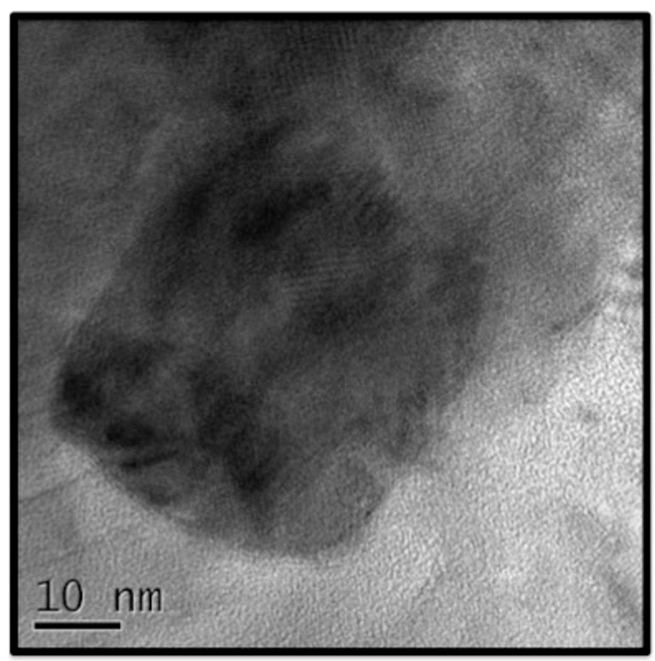

Fig. 6. Amorphous and crystalline mixed carbonaceous matter containing As and Pb.

quantitative analyses by ESD are approximate because elements of lower atomic weights such as H, F, O, and C, among many others, are poor electron emitters (Silva et al., 2010). Consequently, the quantification is not reliable, hence EDS graphs were preferably used in this work.

A total of thirty-five examined particles contained quartz mixed with Al–Fe–Si–K–Ti–O-amorphous phases. In contrast, zircon was the minor abundant mineral found in twenty-two analyzed ultra-fine particles along with some associated amorphous phases of Al, Fe, K, O, Mg, Mn, Si, P, and Ca. The diminution of quartz could

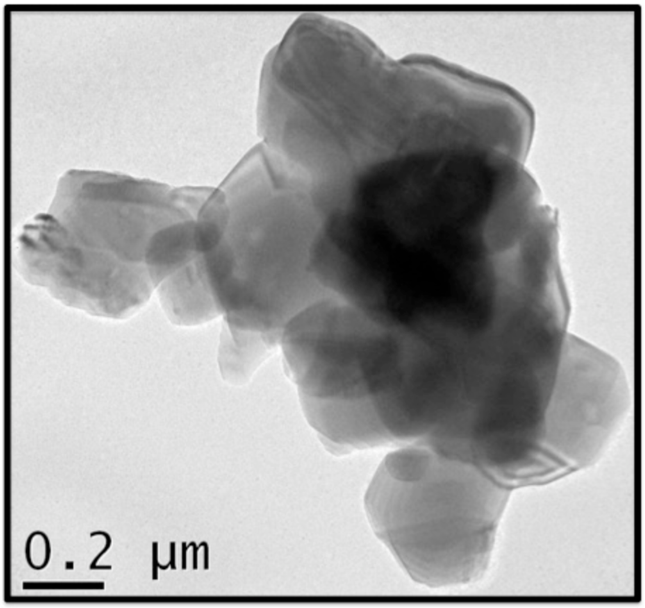

Fig. 7. Typically detected rutile agglomerated.

be associated with the synchronized decrease in the silicon proportions in SS. In addition, it is noticed that the aggregation of NPs and ultrafine particles (10–60 μm) in SS increased through aging, thus PHEs moved to the carbonaceous areas of SS. The sorption of oxy-anionic species and PHEs cations in SS needs to be analyzed in future studies. Moreover, the current work validates the preservation of arsenic, cadmium, chromium, lead, mercury, uranium, zircon, and others PHEs by SS.

Iron nanominerals (sulfates and hydr/oxides) often originate in the natural environment and play a central role in the auto-recuperation of environmental contamination. Several Fe-nanoparticles comprising of Cd, Co, Cr, Cu, Hg, Pb and others PHEs were detected by the electron beam method (e.g. Fig. 5). It is considered that human activities are responsible for the excess amount of chromium in SS (Kazakis et al., 2018). Coal mining, petroleum refining, fabrication of steel, leather tanning, fertilizer and dye manufacturing are the main sources of chromium in the Magdalena SS. In addition, it is believed that the intensity of chromium (VI) in the Magdalena basin is increased by numerous natural progressions, such as the suspension of chromium-containing NPs, the adsorption of OMs, the adsorption and precipitation of dioxides onto SS, and the ion exchange reactions (Kazakis et al., 2018).

It is noticed that arsenic, cadmium, copper, lead, and mercury (e.g. Fig. 6) have larger propensity to be stored in water as compared to other PHEs. PHEs in SS are generally produced due to human activities, such as the uncontrolled development of refinery and coal industries, residual water irrigation and, the extreme utilization of agrochemical (Civeira et al., 2016). The Magdalena River Delta area is the most industrialized and populated zone in Colombia hence, water contamination is one of the greatest disturbing ecological questions in this territory (Tejeda-Benitez et al., 2016). The Barranquilla city has experienced fast change from an agronomy-founded economy over the past few years.

Rutile and anatase TiO_2 NPs (size of 5–263 nm) can change the geomobility and the ecotoxicity of pollutants in the SS because of their size, excellent reactivity, large surface area, and remarkable adsorption ability (Domingos et al., 2009). Therefore, a detailed study on the influences of TiO_2 NPs on PHEs is necessary to identify the probable ecological hazards of these particles (Fan et al., 2018). The excessive application of TiO_2 NPs in Colombia has increased the bioavailability of arsenic, cadmium, copper, chromium, and lead in the Magdalena SS (Fig. 7). Furthermore, the geochemical individualities of sediment-water interface and other organisms manifest dangerous threats to the Magdalena SS. Titanium dioxide (TiO_2) (anatase and rutile) nanoparticles reportedly trigger adverse responses in vulnerable animals. The anatase TiO_2 nanoparticles produce more free radicals than rutile form and, therefore, higher toxicity than the rutile TiO_2 nanoparticle. The anatase TiO_2 nanoparticles are one of the main sources of inflammation and cytotoxicity (Nordin et al., 2018).

The geochemical variations in hazardous elements abundance could be attributed to: i) anthropogenic inputs such as mining, industrial/domestic discharges, etc., ii) a difference in hydrodynamic conditions regulated by fresh-water flow after the storms, and iii) a dispersal of sediments into the Magdalena river after the strong storms.

3.3. *Anthropogenic influences*

Human activities are mainly responsible for the excessive presence of PHEs in the Magdalena SS (Wall et al., 2015). Mohanty et al. (2016) found that NPs can act as transporters to expedite the transference of adsorbed PHEs through porous channels (Xia et al., 2018). Moreover, in order to detect the contamination effects of PHEs, an elaborate understanding of the characteristics of transportable NPs and their adsorbed PHEs is important. The use of advanced analytical tools in this experiment helped in determining the hazardous effects of PHEs and NPs on SS. The application of the wet-geochemical method and microscopy technique revealed the formation mechanism of NPs as well as the mobility of PHEs in SS.

4. Conclusions

The SS samples were found to be coarser in the dry period in comparison to the rainy season because the sediments might be created from river bank erosion instead of being delivered from upstream catchments. Although the combination of numerous sampling methods manifested excellent results, it still possesses some ambiguities.

The ejection of domestic, manufacturing and agronomic wastes to the Magdalena River causes hazardous threats to the aqueous environment of Colombia. The current study depicted the formation of NP containing PHEs in the Magdalena river basin during a flood, thus resulting in leaching of PHE pollutants. The formation of dispersed NPs was closely related to the mechanism of NPs growth from leached phases. Therefore, our findings could contribute to better understand the formation of NPs in the Magdalena river basin and their consequences on the geomobility of PHEs. Supplementary studies related to the weathering process of the land surface can elucidate the weathering processes within Magdalena SS. The ecological analysis of anthropogenic NPs is necessary to comprehend the ecology of coastal areas.

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